FORM PTO-1390 U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY'S DOCKET NUMBER: 5648

U.S. APPLICATION NO: 10/049860

INTERNATIONAL APPLICATION NO	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/GB00/03234	21 August 2000	19 August 1999

TITLE OF INVENTION: PROCESS FOR RECYCLING IONIC LIQUIDS

APPLICANT(S) FOR DO/EO/US

A. J. JEAPES, R. C. THIED, Kenneth Richard SEDDON, W. R. PITNER, D.W. ROONEY, Justine E. HATTER and T. Welton

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31)
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
   a. ☒ is attached hereto (required only if not communicated by the International Bureau).
   b. ☐ has been communicated by the International Bureau.
   c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2))
   a. ☐ is attached hereto.
   b. ☐ has been previously submitted under 35 U S.C. 154(d)(4).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   a. ☐ are attached hereto (required only if not communicated by the International Bureau).
   b. ☐ have been communicated by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   d. ☐ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)) (Unexecuted)
10. ☐ An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 20 below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☐ A FIRST preliminary amendment.
14. ☐ A SECOND or SUBSEQUENT preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☒ Other items of information:
    International Preliminary Examination Report With Amended Claims 18-21.
    Application Data Sheet.

U.S. APPLICATION NO. 10/049860	INTERNATIONAL APPLICATION NO. PCT/GB00/03234	ATTORNEY'S DOCKET NO. 5648

21. ☒ The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492(a)(1)-(5)):

Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1 445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO.. .. .. $1,040

International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO..... ....$ 890

International preliminary examination fee (37 CFR 1 482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO.. .....$ 740

International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4)..... . . ..$ 710

International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4).....$ 100

ENTER APPROPRIATE BASIC FEE AMOUNT =

CALCULATIONS FOR PTO USE ONLY

$ 890.00

Surcharge of $130.00 for furnishing the oath or declaration later than ☐ 20 ☒ 30 months from the earliest claimed priority date (37 CFR 1.492(e)).				$ 130 00	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	$	
Total claims	24 - 20 =	4	x $ 18 00	$ 72 00	
Independent claims	02 - 03 =	0	x $ 84.00	$ 0.00	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+$280.00	$ 280.00	
TOTAL OF ABOVE CALCULATIONS =				$1,372 00	
☐ Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.				$ 0.00	
SUBTOTAL =				$1,372.00	
☐ Processing fee of $130 for furnishing the English translation later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(f)).				$ 0.00	
TOTAL NATIONAL FEE =				$1,372.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) $40.00 per property				$ 0.00	
TOTAL FEES ENCLOSED =				$1,372.00	
				Amount to be refunded:	$
				charged:	$

a. ☒ A check in the amount of $ 1,372.00 to cover the above fees is enclosed.

b. ☐ Please charge my Deposit Account No. _____ in the amount of $ _____ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 02-3690. A duplicate copy of this sheet is enclosed.

d. ☐ Fees are to be charged to a credit card. **WARNING**: Information on this form may become public. Credit card information should not be included on this form. Provide credit card information and authorization on PTO-2038.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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Date: February 19, 2002

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PROCESS FOR RECYCLING IONIC LIQUIDS

FIELD OF THE INVENTION

This invention relates to processes for treating spent ionic liquids. Reference will be made hereinafter to ionic liquids which have been used in the reprocessing of nuclear fuels but it should be understood that the invention has application in all fields of ionic liquids technology, including applications outside the nuclear industry.

BACKGROUND OF THE INVENTION

Ionic liquids free of molecular solvents were first disclosed by Hurley and Wier in a series of US patents (24446331, 2446339, 2446350). In general terms an ionic liquid is a salt, a mixtures of salts, or a mixture of components which produce a salt or salts, which melts below or just above room temperature. (As used herein, the term "salt" means an entity comprising entirely of cationic and anionic species). Such liquids are known as "ionic liquids" although this term is sometimes used for salts which melt at relatively high temperatures. In this specification, the term "ionic liquid" refers to a salt which melts at a temperature of up to 100°C.

Co-pending patent application PCT/GB99/00246 discloses a method for reprocessing spent nuclear fuel which comprises dissolving the spent fuel or constituent parts of the spent fuel in an ionic liquid to substantially separate fissile material from other components of irradiated fuel. Also disclosed is the subsequent treatment of the resulting ionic liquor, either by solvent extraction or electrochemical treatment to recover the dissolved uranium or plutonium.

In addition to fissile material spent ionic liquid also contains fission products which have been dissolved together with the uranium. Considerable effort has been focused on the development of a cost effective route for the removal of these contaminants, subsequent to the separation of the uranium from the mixture, in a form suitable for conversion into a stable waste form.

Contaminant removal may be achieved by any one or a combination of a number of different routes. Selected fission products such as plutonium can be electrochemically extracted through the application of a suitable potential. The disadvantage of this is that electrolysis is a costly process, especially when performed on an industrial scale.

Fission products may alternatively or additionally be extracted from the ionic liquid by the addition of an organic solvent. Addition of an organic species results in the precipitation of fission product compounds. The precipitate can be filtered from the ionic liquid and calcined to convert to oxide prior to a vitrification step. Cleaned ionic liquid can then be recycled to the electrorefiner.

Combustion is another option for removal of contaminants, wherein spent ionic liquid is heated to temperatures in excess of 200°C. The disadvantage of this process can be measured in economic terms: Combustion of imidazolium salts results in the production of carbon dioxide, water and a mixture of nitrogen oxides (NO_x). This means that the ionic liquid cannot be recycled and is in fact destroyed.

Since one of the most significant costs in industry will be that of the requisite raw materials, any process which helps to minimise these costs through recycling is clearly a more viable option.

STATEMENT OF INVENTION

According to the present invention there is provided a method for treating a spent ionic liquid composition comprising an ionic liquid and contaminant, the method comprising heating said ionic liquid to form a partial decomposition product thereof, separating said product from said contaminant and reacting the separated product with a reactant to regenerate said ionic liquid.

When 1-methyl-3-ethylimidazolium chloride is heated under reduced pressure, the ionic liquid partially decomposes to give 1-methylimidizole, 1-ethylimidizole,

chloromethane and chloroethane. In the initial experiments the thermolysis products were left for two days at room temperature and re-acted to produce a mixture of 1,3-dimethylimidazolium chloride, 1,3-diethylimidazolium chloride, and 1-methyl-3-ethylimidazolium chloride. These results are surprising because thermal decomposition was previously considered to destroy any potential for recycling of the ionic liquid. In the light of these experiments however thermal decomposition for the cleaning of ionic liquids is now an economically viable alternative.

Preferably the decomposition product is separated together with at least one other decomposition product from the contaminants. Separation is effected by volatilisation during the heating process. In one embodiment of the invention the partial decomposition product is separated from the at least one other decomposition product by distillation.

Preferably the ionic liquid is heated under reduced pressure at or below 2mmHg at a temperature ranging from 200°C to 300°C or more preferably from 220°C to 250°C.

DETAILED DESCRIPTION OF THE INVENTION

Spent ionic liquid, for example 1-methyl-3-ethylimidazolium chloride, is contaminated with fission products, possibly organic radiolysis products, and will require treatment in order to recover the fission products in a form suitable for conversion into a stable waste form.

In a method according to the present invention, the ionic liquid is heated under reduced pressure to a temperature within the range of 200°C to 300°C. In a preferred embodiment 1-methyl-3-ethylimidazolium is heated at a temperature from 220°C to 250°C for 2-3 hours at pressures below 2mmHg. These conditions allow for only partial decomposition of the ionic liquid and therefore the recovery of as many useful component compounds of the ionic liquid as possible. It should be understood that the conditions for partial decomposition will vary for differently substituted imidazolium salts and for different mixtures thereof .

Thermolysis of the ionic liquid results in the evolution of volatile thermolysis products. In the case of 1-methyl-3-ethylimidazolium chloride these are 1-methylimidizole, 1-ethylimidizole, chloromethane and chloroethane. Chloroethane then decomposes to hydrogen chloride and ethene.

Volatile products can be collected in a cold trap and subsequent distillation of the mixture allows for separation of the components. The most volatile components are hydrogen chloride and ethene. Hydrogen chloride can be scrubbed from the system using a hydroxide scrubber and ethene can either be bottled or burnt as a by-product.

1-methylimidizole and 1-ethylimidizole can be separated by distillation to allow for regeneration of the specific ionic liquid 1-methyl-3-ethylimidazolium chloride. It should be understood that separation is not required for processes which operate on a mixture of 1-methyl-3-ethylimidazolium chloride, 1,3-dimethylimidazolium chloride and 1,3-diethylimidazolium chloride.

If the specific ionic liquid 1-methyl-3-ethylimidazolium chloride is required, separated 1-methylimidizole is reacted with chloroethane so as to regenerate the original ionic liquid 1-methyl-3-ethylimidazolium chloride. Because some chloroethane is lost through its decomposition, fresh chloroethane is added to fully regenerate the ionic liquid. 1-ethylimidazole is reacted with chloromethane to regenerate the ionic liquid 1-methyl-3-ethylimidazolium chloride.

In a preferred embodiment of the invention the fission product residue is calcined to convert it into a stable waste form. In an alternative embodiment fission product residue is reacted with boric acid to convert it into a suitable form for disposal.

CLAIMS

1. A method for treating a spent ionic liquid composition comprising an ionic liquid and contaminants, the method comprising heating said ionic liquid to form a partial decomposition product thereof, separating said product from said contaminants and reacting the separated product with a reactant to regenerate said ionic liquid.

2. A method according to claim 1 wherein said partial decomposition product is separated together with at least one other decomposition product from said contaminants.

3. A method according to claim 2 or claim 3 wherein said partial decomposition product is separated from the at least one other decomposition product by distillation.

4. A method according to any of the preceding claims wherein said partial decomposition product is reacted with at least one other decomposition produced to regenerate said ionic liquid.

5. A method according to any of the preceding claims wherein said separation is effected by volatilisation during the heating process.

6. A method according to any preceding claim wherein the ionic liquid is 1-methyl-3-ethylimidazolium chloride.

7. A method according to claim 6 wherein the partial decomposition product is a mixture of 1-methylimidizole, 1-ethylimidizole, chloromethane and chloroethane.

8. A method according to claim 7 wherein 1-methylimidizole is reacted with chloroethane to regenerate 1-methyl-3-ethylimidazolium chloride.

9. A method according to claim 5 wherein the partial decomposition product is 1-ethylimidizole.

10. A method according to claim 9 wherein 1-ethylimidizole is reacted with chloromethane to regenerate 1-methyl-3-ethylimidazolium chloride.

11. A method according to any preceding claim wherein the ionic liquid is heated under reduced pressure.

12. A method according to claim 11 wherein the pressure is at or below 2mmHg.

13. A method according to any preceding claim wherein the ionic liquid is heated to a temperature from 200 to 300°C.

14. A method according to claim 13 wherein the ionic liquid is heated to a temperature from 220 to 250°C.

15. A method according to any of claims 3 to 14 wherein volatile products resulting from volatilisation of the ionic liquid are collected in a cold trap.

16. A method according to any preceding claim wherein hydrogen chloride is produced by heating said ionic liquid and is scrubbed from the system using a hydroxide scrubber.

17. A method according to any preceding claim wherein ethene is produced by heating said ionic liquid.

18. A method according to claim 17 wherein said ethene is bottled or burnt as a by product.

19. A process for the reprocessing of nuclear fuel and the treatment of salt wastes contaminated with fission products, the process including a method of any preceding claim.

20. A process according to claim 19 wherein the fission product contaminant residue is separated and calcined before disposal.

21. A process according to claim 19 wherein the fission product contaminant residue is reacted with boric acid before disposal.

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(81) Designated States *(national)*: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

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For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette

WO 01/15175 A2

(54) Title: PROCESS FOR RECYCLING IONIC LIQUIDS

(57) Abstract: A method for treating a spent ionic liquid composition includes heating the composition to form a partial decomposition product thereof. The product is separated from composition contaminants and the separated product is reacted with a reactant to regenerate the ionic liquid.

COMBINED DECLARATION AND POWER OF ATTORNEY
FOR PATENT APPLICATION

Docket No. 5648

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled PROCESS FOR RECYCLING IONIC LIQUIDS

,

the specification of which is attached hereto unless the following box is checked:

[X] was filed on 21 August 2000 as United States Application Number or PCT International Application Number PCT/GB00/03234 and was amended on 09 November 2001 (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR §1.56.

I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application(s)			Priority Claimed
PCT/GB00/03234 (Number)	PCT (Country)	21 August 2000 (Day/Month/Year Filed)	Yes [X] No [ ]
9919606.5 (Number)	Great Britain (Country)	19 August 1999 (Day/Month/Year Filed)	Yes [X] No [ ]

COMBINED DECLARATION & POWER OF ATTORNEY

Docket No. 5648

I hereby claim the benefit under 35 U.S.C. §119(e) of any United States provisional application(s) listed below.

(Application Number)	(Filing Date)
(Application Number)	(Filing Date)

I hereby claim the benefit under 35 U.S.C. §120 of any United States application(s), or §365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. §112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR §1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

(Application No.)	(Filing Date)	(Status-patented, pending, abandoned)
(Application No.)	(Filing Date)	(Status-patented, pending, abandoned)

I (we) hereby appoint the following attorney with full power of substitution to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

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COMBINED DECLARATION & POWER OF ATTORNEY

Docket No. 5648

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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1 0

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COMBINED DECLARATION & POWER OF ATTORNEY

Docket No. 5648

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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